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IS 15396 (2003): Chemical Analysis of
Ferro-Silicon-Zirconium Alloys [MTD 5: Ferro Alloys]



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“Knowledge is such a treasure which cannot be stolen”

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भारतीय मानक
फेरोसिलिकॉन-जिरकोनियम मिश्रधातुओं का
रासायनिक विश्लेषण

Indian Standard
CHEMICAL ANALYSIS OF FERRO-SILICON-
ZIRCONIUM ALLOYS

ICS 77.100

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FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Chemical Analysis of Ferrous Metals Sectional Committee had been approved by the Metallurgical Engineering Division Council.

Ferro-silicon-zirconium alloys are master alloys which are required for making additions of silicon and especially zirconium to prepare iron base alloys. Zirconium is for refinement and grain size improvement and silicon for imparting strength to the alloys.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

CHEMICAL ANALYSIS OF FERRO-SILICON-ZIRCONIUM ALLOYS

1 SCOPE

This standard covers the methods of chemical analysis of various constituents of ferro-silicon-zirconium alloys, general composition for which is given below:

<i>Composition</i>	<i>Percent</i>
Silicon	30 to 50
Iron	60 to 70
Zirconium	12.0 to 40.0

2 REFERENCES

The standard listed below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<i>IS No.</i>	<i>Title</i>
264 : 1976	Nitric acid (<i>second revision</i>)
265 : 1993	Hydrochloric acid (<i>fourth revision</i>)
1070 : 1992	Reagent grade water (<i>third revision</i>)
1472 : 1977	Methods of sampling of ferro alloys for determination of chemical composition (<i>first revision</i>)

3 SAMPLING

The sample shall be drawn and prepared as per the method given in IS 1472.

4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and distilled water (*see* IS 1070) shall be employed for the test.

5 DETERMINATION OF SILICON BY THE GRAVIMETRIC METHOD

5.1 Outline of the Method

The sample is decomposed by fusion with sodium peroxide. Silica is separated by dehydration with hydrochloric acid. The solution is filtered. Silica is ignited and weighed. It is then volatilized with hydrofluoric acid. The residue is ignited and weighed. The loss in mass represent silica.

5.2 Reagents

5.2.1 Sodium Peroxide, solid.

5.2.2 Hydrochloric Acid, rd = 1.16 (conforming to IS 265).

5.2.3 Dilute Hydrochloric Acid, (1:1) and (5:95).

5.2.4 Hydrofluoric Acid, 48 percent.

5.3 Apparatus

5.3.1 Iron/Nickel Crucible, 30 ml capacity.

5.3.2 PTFE Beakers, 250 ml capacity.

5.3.3 Platinum Crucible, 30 ml capacity.

5.4 Procedure

5.4.1 Transfer 0.5 g of the sample to a 30 ml iron/nickel (preferable) crucible. Add 5 g of sodium peroxide. Mix thoroughly using an iron/nickel platinum rod and transfer any adhering particles on the rod to the crucible by gently tapping.

5.4.2 Cover the mixture with a layer of 2 g sodium peroxide. Heat the crucible on a hot plate for 10-15 min to expel any moisture. Carefully fuse over a low flame muffle furnace, slowly rotating the crucible, until the contents have melted. Increase the temperature to bright redness for a minute.

5.4.3 Remove the crucible from the flame, cover and allow to cool. Tap the crucible gently and transfer it to a 250 ml stainless steel PTFE beaker. Add 50 ml water cautiously. When the reaction ceases, remove the crucible and rinse with water, adding the washings to the beaker.

5.4.4 Acidify with hydrochloric acid (1:1), add another 50 ml hydrochloric acid (1:1). Transfer the solution to a 250 ml glass beaker. Wash the stainless steel/PTFE beaker with hydrochloric acid (1:1), transferring the washings to the glass beaker.

5.4.5 Evaporate to dryness on a water bath when the residue is dry, allow to cool. Add 20 ml hydrochloric acid, stir well cover the beaker, and heat for a few minutes. Dilute with 200 ml of hot water, digest for a few minutes and filter through a filter paper, containing some filter pulp. Wash with hot hydrochloric acid (5 : 95) eight times and then thoroughly with hot water. Reserve the paper and residue.

5.4.6 Evaporate the filtrate to dryness and bake to 110°C for 1 h. Cool, add 20 ml hydrochloric acid, digest on a water bath for 10 min. Add 20 ml water, stir well, filter and wash with hot hydrochloric acid (5:95) eight times and then with hot water, till free from chloride. Preserve the filtrate, after making it up to 250 ml.

5.4.7 Place the filter paper with residue in a 50 ml platinum crucible, heat slowly to remove the water, raise the heat to char the paper and finally ignite at 1 100°C, after covering the crucible. Cool in a desiccator. Weigh and again ignite at 110°C for 10 min and check for constancy in mass.

Add 2 to 3 ml sulphuric acid (1:1) to moisten the residue, followed by 5 ml hydrofluoric acid.

Evaporate to dryness, ignite at 1 100°C and weigh.

5.5 Blank

Carry out a blank determination following the procedure, but without the sample.

5.6 Calculation

$$\text{Silicon, percent by mass} = \frac{[(A-B)-C] \times 0.4674 \times 100}{D}$$

where

- A = mass, in g, of the crucible before hydrofluorization;
- B = mass, in g, of the crucible after hydrofluorization;
- C = correction for the blank, in g; and
- D = mass, in g, of the sample taken.

6 DETERMINATION OF IRON

6.1 Outline of the Method

In the filtrate, after removal of silica, iron is reduced with a slight excess of stannous chloride. The excess is oxidized by addition of mercuric chloride solution. After addition of sulphuric-phosphoric acid mixture, ferrous iron is titrated with potassium dichromate, using diphenylamine sulphonate, as indicator.

6.2 Reagents

6.2.1 Hydrochloric Acid, rd = 1.16 (conforming to IS 265).

6.2.2 Dilute Hydrochloric Acid, (1:1) and (1:9).

6.2.3 Nitric Acid, rd = 1.42 (conforming to IS 264).

6.2.4 Mercuric Chloride Solution, saturated.

6.2.5 Stannous Chloride (10 percent) — Dissolve 100 g of stannous chloride in 500 ml hydrochloric acid, dilute to 1 litre. Keep in a dark closed bottle,

after addition of a small quantity of tin.

6.2.6 Dilute Sulphuric Acid, (1:19).

6.2.7 Ferrous Ammonium Sulphate Solution (0.10 N) — Dissolve 40 g of $\text{FeSO}_4(\text{NH}_4)_2 \cdot \text{SO}_4 \cdot 6\text{H}_2\text{O}$ in dilute sulphuric acid. Transfer to a 1 litre flask and dilute to volume with the same acid.

Pipette out 50 ml of the solution in a 600 ml beaker, dilute to 400 ml with water. Add 20 ml of acid mixture and 5 drops of indicator. Titrate with hot dichromate to purple end point.

6.2.8 Potassium Dichromate Standard Solution (0.1 N) — Transfer 4.901 5 g of $\text{K}_2\text{Cr}_2\text{O}_7$, previously dried at 110°C to a 1 litre flask. Dissolve in water and dilute to the mark.

6.2.9 Phosphoric Acid (H_3PO_4) and Sulphuric Acid (H_2SO_4)

6.2.10 Diphenylamine Sulphonate

6.3 Procedure

6.3.1 Pipette out an aliquot, containing approximately 50 to 100 mg iron, from the solution preserved 10 under 5.4.6 and transfer to a 600 ml beaker. Add stannous chloride solution, dropwise till the yellow colour of the solution disappears. Add 3 drops in excess. Dilute to 200 ml with water and cool to 10°C. Add 15 ml of saturated mercuric chloride solution, all at once. Mix well and keep on the water bath for 3 to 5 min.

6.3.2 Add 20 ml of acid mixture and 5 drops of indicator solution. Dilute to 400 ml and titrate with standard potassium dichromate solution to a distinct purple end point.

6.3.3 Blank

Determine the blank value of the reagents, using the same amount of all reagents and following the procedure. Before titration with potassium dichromate solution, add 1.0 ml of ferrous ammonium sulphate solution. In another beaker, place 400 ml of cold dilute sulphuric acid and add 1 ml of ferrous ammonium sulphate solution. Add 20 ml of acid mixture and 5 drops of indicator. Titrate with potassium dichromate solution. Record this titration and subtract the value obtained from the blank, to get the corrected blank.

6.4 Calculation

$$\text{Iron, percent by mass} = \frac{(A-B) \times C}{D} \times 100$$

where

- A = volume, in ml, of potassium dichromate solution required for titration of sample;

- B = volume, in ml, of potassium dichromate solution required for titration of blank;
 C = iron equivalent of potassium dichromate solution, in g per ml; and
 D = mass, in g, of sample taken for titration;

7 DETERMINATION OF ZIRCONIUM BY GRAVIMETRIC METHOD

7.1 Outline of the Method

In the filtrate, after removal of silica, zirconium is precipitated as the mandelate, by the addition of mandelic acid. Zirconium mandelate, thus obtained, is ignited and weighed as the oxide, ZrO_2 .

7.2 Reagents

7.2.1 Dilute Ammonia, (1:40) and (1:9).

7.2.2 Hydrochloric Acid, rd = 1.16 (conforming to IS 265).

7.2.3 Mandelic Acid (15 percent) — Dissolve 15 g of mandelic acid in water and make up to 100 ml.

7.2.4 Mandelic Acid Wash Solution — Dissolve 10 g of mandelic acid in 100 ml hydrochloric acid.

7.3 Procedure

7.3.1 Pipette out 50 ml of the filtrate, after removal of silica, as given in 5.4.6 to a 250 ml beaker. Adjust the acidity to 4 from 6 in hydrochloric acid. Add 35 ml of mandelic acid solution and dilute to 150 ml. Digest the solution for 1 h on a water bath, until the precipitate settles down, leaving a clear supernatant liquid.

7.3.2 Filter through a close textured filter paper and wash 3 times with 10 ml portions of cold mandelic acid wash solution.

7.3.3 Dissolve the precipitate on the filter using 30 ml ammonia (1:4) and collect the solution in the same beaker in which precipitation was carried out. Repeat this operation twice, using each time, 30 ml ammonia (1:4). Finally give 2 washings to the filter paper with 5 ml of ammonia (1:9). See that the ammonia solution comes in contact with all sides of the beaker and that any adhering precipitate is brought into solution.

7.3.4 Add 25 ml mandelic acid solution, 3 drops of methyl red indicator, and neutralize with hydrochloric acid. Add 10 ml of hydrochloric acid, in excess, stir and allow to digest on the water bath for 1 h.

7.3.5 Filter warm, through a close textured filter paper and wash the precipitate six times with 10 ml of 10 percent cold mandelic acid wash solution.

7.3.6 Transfer the paper and precipitate to a tared platinum crucible, dry, char and finally heat to 1 000°C. Keep at this temperature for 30 min. Cool and weigh as ZrO_2 .

7.4 Blank

Carry out a blank determination following the same procedure, but without sample aliquot(s).

7.5 Calculation

$$\text{Zirconium, percent by mass} = \frac{(A - B) \times 0.7403}{C} \times 100$$

where

A = mass, in g, of ZrO_2 obtained from in the sample aliquot;

B = mass, in g, of ZrO_2 in g in the blank; and

C = mass, in g, of sample in the aliquot taken.

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This Indian Standard has been developed from Doc : No. MTD 2 (4237).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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